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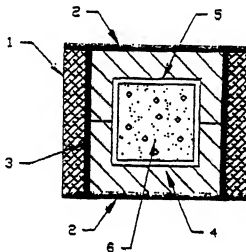
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(57) Abstract

A compact composed of 70 to 95 diamond or cubic boron nitride (or other superhard materials) and which is free of conventional binders, catalysts, and/or solvents and has a thermal conductivity of at least 800 watts/m² K is prepared by subjecting a body (5) consisting essentially of particulate diamond or other superhard materials (6) to a HPHT (High Pressure/High Temperature) step, in which the pressure applied to the body is substantially uniform. The diamond compact is then subjected to Chemical Vapor Deposition (CVD) of the same or different superhard material to infiltrate the interstices of the HPHT compact. The CVD coating can have up to about 2,000 watts/m² K. The compact after both the HPHT and CVD steps substantially retains the thermal and chemical properties of the superhard materials from which it is made.

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DIAMOND COMPACT

FIELD OF INVENTION

5 The present invention is directed to a novel diamond compact and a method of making the same. More particularly, it is directed to a novel diamond compact having many of the properties of single crystal diamond, and which has been made without the use of the binders, 10 such as cobalt, iron, nickel or silicon, which are used in prior art procedures. The method employed involves compression of particulate diamond under high temperature and pressure (HPHT) and preferably followed by a diamond chemical vapor deposition (CVD) step.

15

BACKGROUND OF THE INVENTION

Diamond, diamond compacts or compacts made of cubic boron nitride (CBN) are widely used in industry as cutting 20 elements, drilling bits, machining and woodworking tools, and as heat sinks. Conventional techniques used to make these compacts include forming a bonded diamond compact. These techniques involve sintering a layer of individual diamond particles on a substrate, such as cobalt-tungsten 25 carbide, under conditions of high pressure and high temperature in the presence of a binder such as cobalt. For example, cobalt has been used both as a catalyst for diamond-to-diamond bonding and as a binding agent for binding the compact to a substrate, such as tungsten 30 carbide. The strength and wear resistance of these known sintered compacts degrade at temperatures over 700°C due

to uneven rates of thermal expansion possessed by the diamond and the entrained binder or other inclusions, as well as different rates of thermal expansion possessed by the compact and the substrate. Thus, these compacts do not make good tools for use at high temperatures.

Many other techniques have been proposed and used to make synthetic diamond compacts from particulate diamonds. These techniques include, for example, the use of HPHT processes to consolidate diamond powders in the presence of various sintering aids other than cobalt, such as nickel, iron, and silicon, into monolithic parts for machining tools. Recently a variety of CVD or similar techniques have been proposed as a way to achieve a similar end by coating a hard metal, typically a cobalt-tungsten carbide (Co-WC) tool with a thin layer of diamond to act as the cutting surface.

Typical polycrystalline diamond compacts (PCD) formed from a HPHT process are difficult to shape and fabricate. The shapes and forms that can be manufactured are limited by the available techniques and by economic necessity. The compact made by this invention is easily shaped and fabricated because it is not fully hard after the HPHT step when the bulk of fabrication would be done.

The various CVD processes have not shown much success in diamond-coating hard metal tools due to deleterious interactions with the cobalt binders used in most of these parts. Adhesion and toughness of these coatings have also been a problem due to a lack of mechanical interlock with the underlying substrate. One technique being tried to overcome this problem is to leach out the cobalt binder from the surface matrix prior to CVD. Diamond vapor should then infiltrate the void space, and thereby mechanically lock the coating to the underlying matrix. However, a problem would still exist with the

- internal interface of CVD diamond with the cobalt-containing portion of the hard metal. One patent (U.S. Patent No. 4,882,138) has suggested a technique of avoiding the CVD/cobalt problem by "gluing" together powdered diamond with application of diamond CVD. This proposed approach suffers from the inherent mechanical instability of the powdered diamond precursor.
- A paper by H.D. Stromberg and D.R. Stephens discloses sintering diamond particulate under HPHT conditions. This article was mainly concerned with the hardness of the product. The compact removed from their press tended to fracture and fall apart, so they found that the addition of cobalt or other sintering aids beneficial.
- See "Sintering of Diamond at 1800°-1900°C and 60-65 Kbar", H.D. Stromberg et., Lawrence Radiation Laboratory, Univ. of Calif., Livermore, Dec. 17, 1969 submitted as a manuscript to Journal of The American Ceramic Society.
- The following United States Patents disclose compacts of diamonds or other superhard material: U.S. 5,011,514 to Cho et al. for "Cemented and Cemented/Sintered Superabrasive Polycrystalline Bodies and Methods of Manufacture Thereof"; U.S. 4,882,138 to Pinneo for "Method for Preparation of Diamond Ceramics"; U.S. 5,043,120 to Corrigan for "Process for Preparing Polycrystalline CBN Ceramic Masses"; U.S. 5,011,509 to Frushour for "Composite Compact with a More Thermally Stable Cutting Edge and Method of Making the Same"; U.S. 4,536,442 to Bovenkerk et al. for "Process for Making Diamond and Cubic Boron Nitride Compacts"; U.S. 4,289,503 to Corrigan for "Polycrystalline Cubic Boron Nitride Abrasive and Process for Preparing same in the Absence of a Catalyst"; U.S. 4,248,606 to Bovenkerk et al. for "Supported Diamond"; U.S. 4,985,651 to Ringwood for "Diamond Compacts"; U.S. 5,010,043 to Ringwood for "Production of Diamond Compacts Consisting Essentially of

Diamond Crystals Bonded by Silicon Carbide; and U.S. 5,011,515 to Frushour for "Composite Polycrystalline Diamond Compact with Improved Impact Resistance". These patents are hereby incorporated by reference.

5

SUMMARY OF THE INVENTION

This invention solves the above-cited problems with the use of a HPHT process followed by a CVD process for synthesizing diamond. When the compact resulting from the HPHT process is subjected to a CVD process, the result is a composite material whose properties benefit from the use of both processes. The properties of the constituent elements are not substantially degraded by the inventive process.

In contrast to previous methods, such as that disclosed in U.S. Patent No. 4,882,138, this invention uses a HPHT process to bind or consolidate particulate diamond into a diamond compact. A CVD process can then be used to infiltrate more diamond into the interstices of the diamond composite compact and/or coat the compact with diamond.

The HPHT diamond compact is made by subjecting a body of particulate diamond or other superhard material such as CBN to a pressure of from about 50 to about 75 kilobars for a period of from about 1 to about 30 minutes at a temperature from about 1,000°C to about 1,600°C. The term "diamond in this disclosure, unless otherwise indicated, also refers to cubic boron nitride (CBN).

In order to achieve the diamond compact of this invention, it is essential that pressure be maintained substantially uniform over the entire surface of the particulate diamond body. By using various pressure transfer media such as pyrophyllite, hexagonal boron

nitride (HBN), sodium chloride (NaCl), potassium chloride (KCl), or talc, which act to produce a substantially hydrostatic pressure field around the diamond particulate, any HPHT system, such as anvil/girdle, piston/cylinder or multi-anvil systems, can supply the containment pressures necessary for this stage of the process.

The diamond compact resulting from the HPHT process is a dense polycrystalline aggregate which substantially retains the thermal properties of the precursor particulate diamond. In addition, the coefficient of friction of a polished surface approaches that of single crystal diamond. It has a density of between about 70% and about 95 of diamond. It has a measured thermal conductivity of at least about 800 watts/m²K. The compact without the prior art use of binders has an unexpectedly high thermal conductivity because the thermal conductivity of the precursor material is substantially retained.

By using the HPHT process to form a partially-bonded compact, without cobalt or other sintering aids, the cobalt/CvD problem of the prior art can be avoided. The compact formed by the HPHT step is suitable for the CVD of a dense, locally-epitaxial, polycrystalline diamond layer having substantial or total infiltration into the pores of the compact.

The compact can be formed into many shapes both during and after the HPHT process by various means, such as the use of sacrificial refractory molds during HPHT, or grinding, lapping, laser-cutting, electrical discharge machining (EDM) or etching after HPHT. These methods are inherently less time consuming and use less energy because, after the HPHT step, the compact may not yet be at full hardness and toughness.

The compact can be subjected to a CVD process. The CVD coating penetrates the surface pores of the compact to further enhance the mechanical and thermal properties of the compact without the use of any metal binders or fillers. The resulting composite can range from a CVD diamond surface layer sealing the internal portion of the HPHT compact from the external environments to the near total infiltration of the HPHT compact by CVD diamond producing a completely consolidated ceramic\ceramic type composite of near theoretical density (3.51 g/cc). The thermal conductivity of the diamond compact formed by the HPHT step is further enhanced by the CVD coating, which itself can have a thermal conductivity of up to about 2000 watts/m²K. The CVD infiltration can be of a material not used to form the compact, such as a CBN coating on a diamond compact or a diamond coating on a CBN compact. A diamond compact with a CVD coating, as formed by this invention, has many uses, such as a heat sink, a wire drawing die, or a cutting tool. As a variation on the CVD of a superhard material, the HPHT compact can be infiltrated with a metal to allow bonding to a substrate. Prior to the metal infiltration, the interstices can be altered by CVD plasma etch or deposit. This can change the texture of the surface or alter the near surface porosity to enhance metal infiltration and bonding.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows a conventional capsule to hold the diamond particulate and exert pressure on it,

Figures 2A and 2B show a monolithic heat sink made with this invention as described in Example 5,

Figures 3A, 3B, 3C and 3D show wire drawing dies made according to this invention as described in Example 9,

Figures 4A, 4B, and 4C show nozzles made according to this invention as described in Example 10,

Figure 5 shows the process for making the dies and
5 nozzles of Figures 9 and 10,

Figure 6A and 6B show standard tooling applications of this invention as described in Example 6,

10 Figure 7 shows thrust bearings made according to this invention as described in Example 11,

Figures 8A and 8B show rotary bearings and shaft seals made according to this invention as described in Example
15 12, and

Figure 9 shows a heat pipe made according to this invention as described in Example 14.

20 DETAILED DESCRIPTION

A particulate diamond, synthetic or natural, is compacted under high pressure high temperature (HPHT) conditions in any standard HPHT apparatus such as belt/anvil or
25 piston/cylinder. The resulting compact can be fabricated into any geometric shape (e.g., planar, spherical, round discs, or cylinders).

Figure 1 shows a capsule that is used in these
30 apparatuses so that uniform pressure is exerted on the diamond being compacted. The capsule is defined by conduction disks 2 and heater shell 3. There is an insulation shell 1 surrounding the heater. Within this capsule is placed a pressure transmitting material 4,
35 such as pyrophyllite, NaCl, KCl, KBr or talc. The diamond particulate 6 is placed in a canister 5 which is surrounded by the pressure transmitting material 4. The

canister is made of a refractory metal, such as molybdenum or zirconium, which may act as a gathering agent. An important factor in the degree of bonding that takes place is the cleanliness and purity of the precursor materials including the canister materials and to a lesser extent the other capsule components. This is typically obtained by a combination of acid leaching, solvent cleaning with ultrasonics, and vacuum or hydrogen heat treatment. The specifics of the crude precursors determine the exact nature of this pretreatment and the choices would be evident to those skilled in the art. Variations on the capsule design are known to those skilled in the art. Pressure is then exerted on the capsule by techniques known in the art.

The pressure exerted is between about 50 and about 75 kilobars for between about 1 and about 30 minutes. No catalyst is needed. The temperature is between about 1000°C and about 1600°C. Preferred conditions of pressure, time and temperature are 60 Kbars at 1350°C for 15 minutes.

After the HPHT process, the diamond compact (referred to as the HPHT compact) of this invention substantially retains the thermal conductivity of the precursor particulate diamond. Measured values of 800 watts/m²K have been obtained on compacts made from natural diamond particulate by this invention. The density of the HPHT compact at this point is between about 70% and about 95% of single crystal diamond, *i.e.*, from about 2.75 g/cc to about 3.25 g/cc, as compared to solid single crystal diamond which has a density of about 3.5 g/cc. The density will depend on the particle size distribution of the precursor diamond particulate.

Removal of any carbon or graphitic phase (or other breakdown products of superhard material precursors) that

form during HPHT can be achieved by leaching with chemical systems, such as HCl/HNO_3 , $\text{H}_2\text{SO}_4/\text{HNO}_3$, bromine or bromine/methanol.

- 5 At this point, shaping of the compact is possible by one or more of a number of techniques, such as grinding, lapping, EDMing, laser cutting, or chemical etching. Shaping of the part can also be partly or fully realized during the HPHT process by the use of sacrificial mold
10 parts according to techniques known to those skilled in the art.

- After shaping, the HPHT compact needs to be cleaned of any extraneous matter that may have contaminated it, such as abrasive slurries or cutting fluids, by techniques
15 described above.

- After the diamond compact has been formed by the HPHT step and, if desired, shaped, it can be provided with a
20 diamond CVD coating of either natural isotopic distribution or enriched isotopic distribution employing known CVD procedures. In the CVD process, diamond infiltrates into the interstices of the HPHT compact, giving good adhesion, better mechanical properties and
25 improved thermal conductivity without the use of a binder. This results in a consolidation of the HPHT compact into a ceramic/ceramic type composite with a surface layer which effectively seals the interior of the diamond compact away from the external environment.

- 30 The fabrication of the diamond compact can be influenced by the porosity of the compact, which can be controlled in a number of ways. For example, the particle size distribution (PSD) of the precursor particulate diamond
35 can be varied to adjust the porosity of the compact formed during the HPHT process. As a general rule, a very narrow PSD will give a much more porous structure

than a wide PSD which has been optimized for maximum packing (e.g., particles having diameters ranging from tens of microns to submicron sizes). Particles having sizes of from about 1000 microns to less than about .01 micron can be used as precursors.

The particle shapes of the precursor particle also play a part in the degree of packing. Typically the feedstock produced by micronizing natural or synthetic diamond is irregular and not amenable to shape sorting or accurate size sorting. Crystalline Materials, 2411-A Old Crow Canyon Road, San Ramon, CA, 94583, has developed diamond CVD techniques which can produce a tailored PSD and diamond particles having regular habits, such as cubo-octahedral or dodecahedral. In addition, these particles can be grown as single crystals or as polycrystalline aggregates. The use of compacts made from these particles should show increased thermal stability in use because of the differential chemical stability of crystallographic surfaces with respect to thermal graphitization. The overall rate of graphitization becomes higher as more of the less thermally stable surfaces are exposed. In addition, a more regular habit crystal has a lower surface area to volume ration than a irregularly shaped particle and is, therefore, more thermally stable. The thermal stability is also enhanced by the lack of metallic impurities in the CVD process.

During the HPHT process, choice of the appropriate particulate precursor, for example CVD diamond, natural diamond, synthetic diamond or cubic boron nitride (CBN), and the precursor's constituent parameters, for example, PSD, particle shape, isotopic purity, and impurity content, along with a suitable pressure in the range of 50 to 75 Kilobars, and a suitable temperature in the range of 1,000 to 1,500 °C, will allow targeting of the dimensional, mechanical, and thermal properties of the

HPHT compact. For instance, PSD along with the applied pressure help determine the final density of the compact and the precursor's thermal conductivity puts an upper limit on the thermal conductivity of the HPHT compact.

5

Prior to actual diamond CVD deposition/infiltration, the HPHT compact can be subjected to a cleaning or etching step using CVD plasma, without a carbonaceous feed gas. This will expose fresh diamond surfaces suitable for enhanced epitaxial growth. In addition, this technique can be used to alter the porosity of the part by etching away part of the structure.

Diamond CVD can be implemented with the intention to either infiltrate all or some of the open porosity of the compact. Which route is chosen depends on the end use. If, for example, only the surfaces are sealed, then the process can be quite fast, depending on the CVD technique chosen. This will leave the compact with a large amount of residual closed porosity, which could be used as a containment for a heat transfer medium, as in a heat pipe application. If most or all of the porosity is filled with CVD diamond, then the compact can be viewed as a diamond-to-diamond composite with its performance parameters determined by the relative properties of the HPHT compact and filler (CVD) diamond. Typically, as the properties of the two materials approach one another, the part should behave like a polycrystalline diamond monolith. As they diverge, the compact behavior should become more like a traditional two-component composite, e.g., Co-WC. The CVD step can also be used to extend the HPHT compacts dimension to or beyond the desired final dimensions, or to produce a large monolithic compact from a close packed array of HPHT compacts 21 bonded together by CVD 22, as shown in Figure 2. A further advantage of the diamond CVD process is that, as the compact wears down, it can be refurbished if so desired.

A variation on the CVD step would be to mix two compatible materials, such as diamond and CBN, to produce a true heterogenous composite. A further variation is to infiltrate a metal into the HPHT compact which would act
5 as a binder to a compatible substrate, e.g., cobalt infiltrate which would allow attachment to a cobalt-tungsten carbide hard tool.

The final finishing operation, if needed, can be
10 accomplished by the same or similar techniques as used in the preliminary shaping of the parts, as discussed above, along with techniques for shaping and finishing of PCD, CBN, or single crystal diamond.

15 One preferred embodiment of the diamond compact is one designed to exhibit the highest thermal conductivity. Such a product is obtained by use of both the HPHT and CVD processes, and requires the use of isotopically pure feedstocks for both the HPHT and the CVD processes. The
20 thermal properties of the constituent precursor are degraded little if at all by the HPHT process step. The HPHT product will, therefore, have properties substantially the same as those of the precursor material.

25 Thermal stability of the product is enhanced by minimizing the levels of metallic and other impurities in the feedstocks, for example by using feedstocks generated totally by a CVD process.

30 The end product of the inventive process is a diamond compact that is all diamond without binders or additives and which substantially retains the unique properties of the precursor diamond particulate, such as high
35 electrical resistance, high thermal transfer coefficient, low thermal expansion coefficient and low coefficient of friction.

The compacts made by this invention can be used in any of the known uses for diamond compacts. Their unique properties make them substitutable for any applications which use single crystal diamonds, such as industrial tools and heat sinks. A heat sink made by this invention could either be a HPHT compact alone or a CVD/HPHT composite compact depending on the application. The prior art compacts made using cobalt or other binders are inferior for such uses at high temperatures due to their lower thermal conductivity and stability.

The following examples are illustrative of the invention.

Example 1 HPHT Compact Using Minimal Particle Size Distribution

A diamond particulate having a particle size distribution between about 30 and about 40 microns purchased from Diamond Abrasive Corp. (DAC) under tradename of SND (natural diamond) was used to form a diamond compact under high pressure and high temperature conditions. The diamond particulate distribution has a Gaussian curve with a mean of about 35 microns. This particulate was placed into a pressure capsule such as that shown in Figure 1. The capsule was then subjected to a pressure of about 60 Kbars at about 1350°C for about 15 minutes. The resulting diamond compact was porous with a density between about 70 and about 85% of single crystal diamond. Some degradation of the thermal conductivity from that of the precursor material is expected because of the decrease in the amount of diamond in the compact.

Example 2 HPHT Compact Made Using An Optimal Particle Size Distribution

Diamond particulate having optimal particle size distribution was made from a mixture of particulate diamond within ranges of PSD purchased from DAC. The

- mixture consisted of about 43% of diamond particulate having a PSD range of about 30 to about 40 microns, about 11% of diamond particulate having a PSD range of about 10 to about 20 microns, about 11% of diamond particulate having a PSD range of about 6 to about 12 microns, about 22% of diamond particulate having a PSD range of about 3 to about 6 microns, about 8.5% diamond particulate having a range of about 1 to about 2 microns and about 4.5% of diamond particulate having a PSD range of about 0 to about 0.5 microns. This particulate was placed in a capsule, such as shown in Figure 1, and subjected to a pressure of about 60 Kbars at about 1350°C for about 15 minutes. The resulting diamond compact was less porous than that produced by the process of Example 1 and had a density of about 92% of single crystal diamond, and a thermal conductivity of about 800 watts/m°k.

- The thermal conductivity was determined by measuring the thermal diffusivity of the compact using a Flash Type Thermal Diffusivity apparatus using an exponential heat pulse technique, as described in "Technical Support Package on Computerized Analysis of Thermal Diffusivity Data," A.B. Chmielewski et al., NASA Technical Briefs, Vol. 12, No. 9, Item No. 88 (October 1988). The measured thermal diffusivity was about 1.67 cm²/sec. at 500°C. Thermal conductivity is equal to the density times the specific heat times the diffusivity. At room temperature, the thermal conductivity of single crystal diamond is between about 600 watts/m°K and about 1000 watts/m°K.

- The thermal conductivity of diamond peaks at about 77°K (-173°C) and then decreases as the temperature further increases, substantially leveling off at about 500°K to 600°K (400-500°C). In this example measurements were made at elevated temperatures and thermal conductivity should get better at lower temperatures. The diamond

compact made from particulate having an optimal PSD substantially retained the thermal conductivity of its precursor. An increase in pressure would result in decreased porosity.

5

Example 3 HPHT Compact With A CVD Coating

The compacts produced as described in Examples 1 and 2 were subjected to CVD by ASTex (Applied Science and Technology), Inc. of 35 Cabot Road, Woburn, MA, 01801, whose specific CVD process is proprietary. The CVD fills the porosity of the surface and the near surface volume, the faster the deposition the less perfect the diamond that is deposited. For a high thermal conductive compact, slower deposition is required to get a more pure deposition. The diamond compact made from 30 to 40 micron particulate is very porous and, therefore, there is more for the CVD coating to lock into and fill. For the compact with optimal packing the CVD coating ranges from about 5 to 30 microns in the surface. The CVD coating seals and finishes the surface.

Example 4 Individual Device Heat Sinks

For the highest level thermal conductivity heat sink, an isotopically pure CVD precursor is used for the HPHT step, such as that used in Example 2. Precursor materials can range in thermal conductivity from about 600 watts/m²K to about 3000 watts/m²K, depending on the application. The HPHT step is tuned, as discussed above, to yield a maximum density of about 90% to 95% of single crystal diamond in the diamond compact; i.e., using a pressure at about 60 Kbars and a temperature at about 1350° for about 15 minutes. At this point, the compact can be used as a heat sink due to its high thermal conductivity of 800 watts/m²K, as discussed in Example 2. There has been no degradation of the thermal conductivity

of the precursor diamond particulate. A CVD coating can also increase the total thermal conductivity of the composite by providing a superior surface finish which enhances thermal coupling. The CVD coating should be sufficient to seal the surface porosity allowing 100 of the surface area of the compact to be used for thermal transfer to the bulk. For some applications, a lower density HPHT compact, such as that produced in Example 1, may be desirable to allow infiltration of a greater amount of higher thermal conductivity CVD component into a lower thermal conductivity compact, thereby enhancing the total thermal conductivity of the composite. The CVD coating will increase the coupling ability because the surface is no longer porous and more surface contact is possible. The coating infiltrates into the bulk of the compact making a superior heat sink. The thermal conductivity of the compact with the CVD coating was not measured but it is expected that the overall thermal conductivity will be altered up.

Example 5 Monolithic Heat Sinks

Referring to Figure 2, the same process is followed as in Example 1 except that the component HPHT parts are fabricated to close tolerances to allow for close packing of the parts, assembled into the desired array as shown in Figure 2A, and then subjected to CVD. Close packing of the HPHT parts enables the CVD layer to bridge and fill any residual gaps 20, as shown in Figure 2B. The choice of CVD coating is dependent on the applications for the heat sink. For example, if the composite is bonded to a mechanically stable metal backplate the coating can be about 50 microns thick. If the composite is to be stabilized mechanically by CVD, then the layer can be of greater thickness such as 500 microns.

Example 6 Standard Tooling Applications

A CVD coated compact is produced employing the processes disclosed in Examples 2 and 3 are followed. For the HPHT step, the highest mechanical strength precursor was chosen, such as a diamond precursor with little or no metallic inclusions and substantially free of any mechanical flaws (., highest quality synthetic or natural diamond). The CVD diamond is applied to the cutting side of the tool to infiltrate the residual porosity, further improving the physical properties such as hardness and toughness, as well as enhancing the thermal properties. The HPHT component is then infiltrated elsewhere in its volume with material suitable for bonding to a substrate. The use of the CVD coating results in an enhanced hardness, as evidenced by the fact that it takes two or three times longer to grind or lap down the compact with diamond abrasion, as compared to the HPHT natural diamond compacts alone.

For this example, as well as examples 7 and 8 that follow, the CVD process results in either a partial infiltration of the HPHT compact, see Figure 6a, or a total infiltration of the HPHT compact, see Figure 6b. For partial infiltration, the CVD diamond 60 is deposited on the cutting edge 68 of the compact. CVD then infiltrates and bonds 61 the CVD diamond 60 to the compact 62. Brazing metal or alloy 63 is then infiltrated into and bonded to the HPHT compact 62 on the noncutting edge 70 which is then bonded to a substrate 64 such as cobalt-tungsten carbide. For total infiltration, CVD infiltrates the HPHT compact 65 on the cutting edge 69. The brazing metal or alloy 66 infiltrates the HPHT compact on the noncutting edge 71. The composite is then bonded to a substrate 67, such as tungsten carbide, on the noncutting edge 71.

Example 7 Stand Alone Tooling Application

Stand alone tooling is produced employing the HPHT and CVD processes as discussed in Examples 1, 2 and 3 can be used. For the HPHT step the highest mechanical strength precursor is chosen, as discussed in Example 6. The HPHT compact is then infiltrated with material suitable for stand alone applications, including complete superhard material infiltration using CVD. Such materials include but are not limited to diamond CVD and CBN CVD. As a variation, instead of the CVD step, a metal such as cobalt could be infiltrated as a toughening agent.

Example 8 Thermally Stable Tooling Applications

The HPHT precursor is created from CVD diamond having high intrinsic thermal stability, such as CVD diamond without metallic impurities and, preferably, Crystalline Materials product, using the procedure of Example 1 or Example 2 followed by the procedure disclosed in Examples 3 and 4.

Example 9 Wire Drawing Dies

Referring to Figure 3, a particulate diamond precursor having the highest mechanical and thermally stability 32 is placed around a refractory sacrificial metal core 30 or shell 31. The HPHT step is performed as described in Examples 1 and 2, and then the refractory core is removed by chemical means, such as acid leaching, as shown in Figures 3C and 3D, to form a die of about 5 mm in thickness and about 15 mm in diameter. A CVD coating can be applied, as in Example 3, to fill the residual porosity of the shaped HPHT component to obtain maximum density and strength. The CVD process makes the wire drawing hole a size suitable for finish by standard wire drawing die fabrication techniques. The compact can then be bonded to tungsten carbide or a similar support. Alternatively, the wire drawing hole can be shaped after

the HPHT step by standard means, such as laser drilling or ultrasonic machining.

5 Example 10 Nozzles for Abrasive Slurries, Liquids, or Gas Streams

Employing procedures similar to those described in Example 9, except that the HPHT compact is formed in a shape suitable for a nozzle, nozzles for abrasive
10 slurries. Liquids or gas streams are fabricated as shown in figures 4A, 4B and 4C.

Example 11 Thrust Bearings

15 Employing procedures similar to those described in Examples 1, 2 and 3, and employing shaping techniques as described in Examples 6, 7 and 8, cylindrical diamond compacts with CVD coating are prepared. The resulting compacts are applied to the opposed flat surfaces of a
20 cylindrical metal carrier, to form a thrust bearing as shown in Figure 7. A thrust bearing made of diamond is of great use, as discussed in Polycrystalline Diamond Creates Superior Thrust Bearings, S.C. Horman, Finer Points, pp 15-16, 24, Sept. 1991. This paper discusses
25 PCDS as good thrust bearings, the diamond compacts of this invention are superior as thrust bearings because they are cheaper, easier to fabricate and thermally more stable.

30 Example 12 Shaft Seals / Bearings

Employing procedures similar to those described in Examples 1, 2 and 3, and forming the compact into arcuate segments by shaping techniques, such as those disclosed
35 in Example 9, compacts useful for seals or bearings are made. The compacts are then bonded to a substrate, such as cobalt-tungsten carbide, with the compact forming the seal or bearing face. As Shown in Figures 8A and 8B, the

seal or bearing is a cylindrical compact 81 bonded to a substrate 82 which, in turn is bonded to shaft 80. The seal or bearing may be formed in two segments, as shown in Figure 8A, or in more segments, (e.g., four, as shown in Figure 8B) for ease of application to shaft 80.

Example 13 In Vivo Applications Including Blood Vessel Router Tools or Prosthetic Implants

10 A tool useful for in vivo use is obtained employing the procedures described in Examples 1, 2 and 3. The precursor diamond used in the HPHT step cannot contain any impurities that may leach out into the body, such as cobalt. Further, the precursor should be pure diamond, 15 e.g., purified natural or CVD diamond. The CVD step is used to fill the HPHT component to maximum density and strength for use as a blood vessel router or other tools where chemical and mechanical stability to prevent contamination is a concern. However, if residual 20 porosity is desired to enable the compact to act as an anchor for regrowing tissue, all or portions of the compact can be left uncoated using standard masking techniques.

25 Example 14 Heat pipes

The processes of Examples 1, 2 and 3 are employed to make heat pipes as shown in Figure 9. A HPHT compact 90 is formed to have a porosity allowing for heat transfer 30 fluid. The porosity is optimized depending on the chosen heat transfer fluid and thermal environment. A CVD coating 91 is applied to three sides of the compact as an encapsulant layer. This composite is then bonded to a metal substrate heat sink 92. The heat transfer fluid 35 (not shown) is injected into the porous diamond compact 90 during one of the sealing operations or is injected afterwards through a resealable port via a channel in the metal substrate heat sink or the CVD diamond layer. A

heat pipe works by using the evaporation/condensation cycle of a heat transfer fluid. The fluid is chosen on the basis of the desired operating temperatures of the heat source side (CVD diamond layer) and the heat sink side (metal substrate). The pore volume and size is then chosen so that the liquid phase of the working fluid goes by capillary action, up the surfaces of the porous diamond compact 90 from a cold zone 94 to a hot zone 93 while not impeding the flow of the vapor phase of the working fluid in the reverse direction. The heat pipe cycle can result in thermal transfer rates on the order of 5 to 10 times greater than solid state thermal conduction.

15 Example 15 Ceramic / Ceramic Composites

Examples of these composites are diamond/CBN, diamond/diamond, CBN/CBN and CBN/Diamond. These composites have many applications in industry in harsh environments or for heat engine parts such as piston heads where mechanical, thermal, and chemical stability are extremely desirable.

25 Example 16 Ceramic / Metal Composites

Examples of these composites are diamond or CBN compacts infiltrated with cobalt, iron, nickel, titanium, or silicon.

30 The composites of examples 15 and 16 are fabricated using the techniques of Examples 1, 2 and 3, with variations for specific applications.

35 Example 17 Synthetic Gemstones

The HPHT process of Examples 1 or Example 2, was adjusted (higher temperature, lower pressure) so as to produce

partial graphitization of the natural diamond precursor. The pressure was decreased to about 58 Kbars and the temperature was increased to about 1400°C. The resultant HPHT compact had a blackish cast with somewhat reduced physical and thermal properties. The HPHT compact was formed by cutting, lapping, and polishing into gemstone shapes with surfaces suitable for diamond CVD. The CVD process was applied to give substantial amounts of graphitic phase to achieve a deep black surface color while maintaining a relatively high degree of hardness. The CVD coating had a thickness of between about 15 microns and about 20 microns. The degree of infiltration and the final CVD surface thickness are determined by economic factors and the requirements for polishing the piece to a gemstone finish.

WHAT IS CLAIMED IS:

1. A diamond compact comprising a diamond particulate compacted under high temperature and high pressure conditions in the essential absence of non-diamond binder or sintering aids, and a diamond infiltrate formed by chemical vapor deposition of diamond.
2. The diamond compact of Claim 1, wherein said high pressure is a substantially uniform pressure of between about 50 kilobars and 75 kilobars and said high temperature is between about 1000°C and about 1600°C, and further having a density of between about 2.75 gm/cc and about 3.25 gm/cc.
3. The diamond compact of claim 2, wherein the diamond particulate is synthetic, manmade or natural diamond particulate.
4. The diamond compact of claim 2, wherein the diamond particulate is isotopically pure.
5. The diamond compact of claim 2, wherein the diamond particulate is made from CVD diamond particulate.
6. The diamond compact of claim 2, wherein the particulate is cubic boron nitride (CBN).
7. A process for making a diamond compact comprising,
 - (a) placing diamond particulate substantially free of binders and sintering aids into a capsule,
 - (b) exerting a substantially uniform pressure of between about 50 kilobars and 75 kilobars over entire surface of the diamond particulate in the capsule, at a temperature between about

1000°C and about 1600°C, for a period between about 1 minute and about 30 minutes, and

- (c) infiltrating the compact formed in b) with a chemical vapor deposition of diamond.
8. The process of claim 7, wherein the diamond particulate is chosen from a group consisting of synthetic or manmade diamond particulate, isotopically pure diamond, CVD diamond particulate, cubic boron nitride (CBN), and natural diamond particulate.
 9. A tool comprising the diamond compact of claim 1.
 10. A tool according to claim 9, wherein said tool is a cutting tool, a wire drawing die, a nozzle or an in vivo apparatus.
 11. The process according to claim 7, further comprising,
 - b2) forming the diamond particulate into a desired shape after the pressure is exerted.
 12. The process according to claim 7, further comprising,
 - b2) forming the diamond particulate into a shape while exerting the pressure by using sacrificial refractory molds.
 13. The process according to claim 7, wherein the temperature is preferably at about 1350°C, the pressure is preferably about 60 kilobars and the time is about 15 minutes.

14. A heat sink comprising a diamond compact consisting essentially of particulate diamond compacted under a substantially uniform pressure of between about 50 kilobars and 75 kilobars and a temperature of between about 1000°C and about 1600°C and having a density between about 2.75 g/cc and about 3.25 g/cc and a diamond infiltrate formed by chemical vapor deposition.
15. A synthetic gemstone comprising the diamond compact of claim 1.

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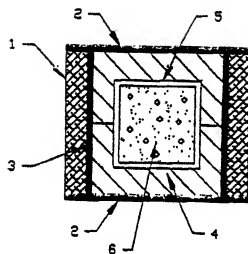


FIGURE 1

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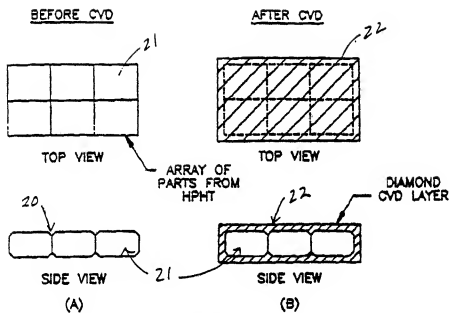
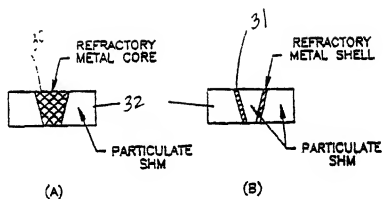


FIGURE 2

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AFTER HPHT PROCESSING AND CHEMICAL LEACHING

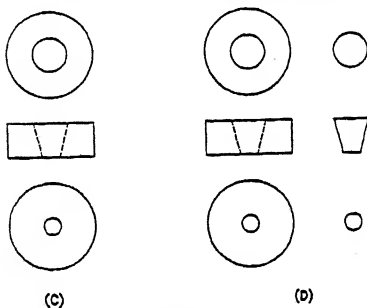


FIGURE 3

VARIOUS NOZZLE PATTERNS

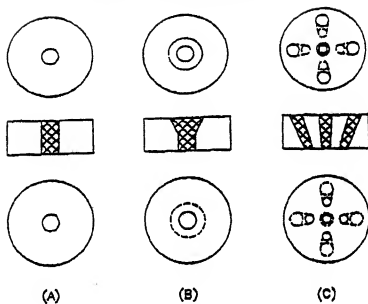


FIGURE 4

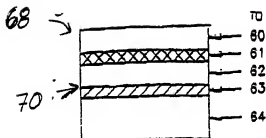


BEFORE CVD

AFTER CVD
AND POLISHING TO
TO FINAL DIMENSION

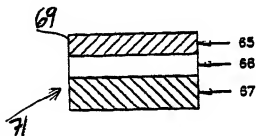
FIGURE 5

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- 60 - CVD DIAMOND
- 61 - CVD INFILTRATED INTO AND BONDED TO HPHT COMPACT
- 62 - COMPACT
- 63 - BRAZING METAL OR ALLOY INFILTRATED INTO AND BONDED TO HPHT COMPACT
- 64 - W_C-C_O OR OTHER SUBSTRATE BONDED TO ABOVE BY BRAZING METAL OR ALLOY OF 63

FIGURE 6A



- 65 - CVD INFILTRATED HPHT COMPACT
- 66 - BRAZING METAL OR ALLOY INFILTRATED HPHT COMPACT
- 67 - W_C-C_O OR OTHER SUBSTRATE BONDED TO ABOVE BY BRAZING METAL OR ALLOY OF 66

FIGURE 6B

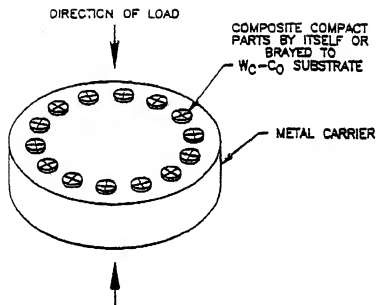
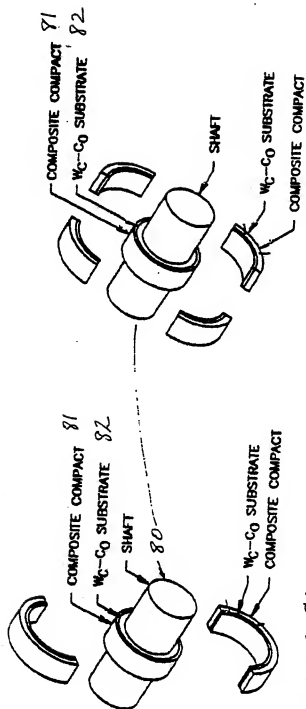
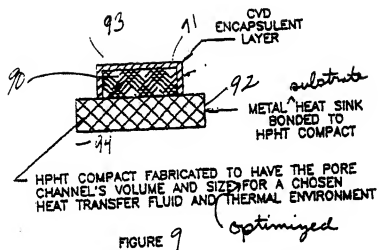


FIGURE 7





INTERNATIONAL SEARCH REPORT

International application No.
PCT/US93/04675**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(5) :B24D 3/00

US CL :51/293,295;427/249;501/86

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 51/293, 295; 427/249; 501/86

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A, 4,248,606, (Bovenkerk et al) February 3, 1981 col. 2, 11.58-65, col. 3, 11.47-57.	1-18
A	US,A, 4,311,490, (Bovenkerk et al), January 19, 1992	1-18
A	US,A, 4,527,998, (Kheneyer), July 9, 1985	1-18
A	US,A, 4,536,442, (Bovenkerk), August 20, 1985	1-18
A	US,A, 4,789,385, (Dyer), December 6, 1988	1-18

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	* later documents published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be part of particular relevance	*X* documents of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

28 JUNE 1993

Date of mailing of the international search report

19 AUG 1993

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US93/04675

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A, 4,874,398, (Ringwood), October 17, 1989 col. 1, ll. 50-61, col. 3, ll. 7-17, col. 4, 1-5, col. 5, ll. 9-34, col. 7, ll. 43-49 and col. 10, ll. 16-23.	1-18
Y	US,A, 4,985,051, (Ringwood), January 15, 1991	1-18
Y	US,A, 5,010,043, (Ringwood) April 23, 1991 col. 5, ll. 35-60 and col. 8, ll.56-60	
y	US,A, 5,011,509, (Frushour), April 30, 1991 col. 1, ll. 65-68 bridged with col. 2, ll. 1-2 and col. 2, ll. 19-30.	1-18
A	US,A, 5,043,120, (Corrigan), August 27, 1991	1-18
A	US,A, 5,106,393, (Ringwood), April 21, 1992	1-18

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US93/04675

B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

APS: Abrasive (and) compact (and) diamond (and) particulate (and) infiltrate (and) high temperature (and) pressure and chemical vapor deposition.